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EXPERIMENTAL INVESTIGATION ON THE EFFECT OF CONNECTION TUBE MATERIAL ON DEW POINT MEASUREMENT IN CALIBRATION INSTRUMENTATION AND UNCERTAINTY EVALUATION

YUN-KYUNG BAE

Industrial & Physical Instrument Center, Industrial Standards Division, Korea Testing Laboratory, Gyeonggi-do, Korea

ABSTRACT

In this paper, the effect of of connection tube material in calibration system on the measurement of dew point temperature for Dew Point Sensors (DPS) and estimation of measurement uncertainty were investigated by means of experimental and statistical technique. The calibrations for the sampling DPSs as the experimental method due to tube materials were performed for over a year. The investigation was focused on variances of correction and deviation of measured dew points indicated by capacitive-aluminum oxide DPSs related to connection tube with stainless steel, copper, PTFE and PVC between -60 ℃ and 10 ℃, the reference dew points. The contributions to the standard uncertainty related to tube material effect were calculated at respective reference dew points to verify the effect of tube material considered an uncertainty component. The whole reference instruments securing traceability were used and calibration procedure were carried out based on standard calibration procedure of Korea Testing Laboratory accredited from ILAC (International Laboratory Accreditation Cooperation) and KOLAS (Korea Laboratory Accreditation Scheme).

KEYWORDS: Dew Point Temperature, Tube Material, Calibration, Measurement Uncertainty, Uncertainty Contribution & Capacitive-Aluminum Oxide Dew Point Sensor

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Original Article

INTRODUCTION

Dew point temperature is a point occurred condensation by air cooled down. The water vapor begins to condense on cold surface under saturation state that air can't absorb water vapor any more. Therefore, the higher a certain space temperature under constant pressure condition, the higher the dew point temperature. It is indispensable parameter determining absolute or relative humidity as a measure of how much water vapor is in a gas. In general, dew point measurement is usually performed in many industrial and meteorological application as well as manufacturing and the package process.

Dew point sensors are used widely in the management of pipeline condition and gas turbine, package process control of micro-electric, medical supplies, the quality of food stuffs and the growth of microorganisms [1-2, 8, 9]. Therefore, the reliability of measurement results for dew point temperature play crucial roles in improvement of product quality and process efficiency and guarantee of the safety of the public.

Most reference Dew Point Sensors (DPS) are classified into chilled mirror types and capacitive-aluminum oxide types, which is widely used to measure water vapor content of a gas [1,3,16-17]. The chilled mirror sensors are used as reference standards in many calibration institues owing to its high accuracy and precision. Those type is usually applied for absolute humidity measurement and determined water vapor content by optically detecting dew or frost formation on the chilled mirror [8-9,16,17]. The capacitive-aluminum oxide sensors are measured by

electrical signal change according to the amount of water vapor contained in the air [3,5]. Those types can measure even the low dew point area that the chilled mirror sensor cannot meausre, have a quick response and are portable. However, those are susceptilble to the infuence of the surrounding environment and have relatively high uncertainty, requiring calibration according to a fixed cycle.

In most of the previous study, the effects of characteristics of dew point generator and various environmental conditions on measurement of dew point temperature have been investigated [10-12]. New dew point measuring method and generator have been introduced [13]. In addition, a research was conducted to apply the environmental factors affecting dew point measurement as measurement uncertainty component [14-15]. Condensation or adsorption rate of water vapor due to tubes with various material properties and geometrical structures has been studied [4-7]. It has been suggested mutual relationship and effect of the environmental condition such as surface shape of material, atmospheric pressure, vapor velocity and structural variance on adsorption or condensation occurred on the surface of material.

Until now, the measurement system and sensors for dew point measurement are connected and used without distinguishing the tube material in the industrial field. In other words, measurement of the dew point temperature has been performed without regard to the characteristic of line material in workplace. This means that the relationship between the measurement result and the tube material is not taken into account. In particular, because of the electrical characteristics of capacitive-aluminum oxide type dew point sensors, measurement errors can easily occur due to changes in various measurement environments. Therefore, the effect of state change in gas flow due to the line material should be considered. Even if the Dew Point Sensor (DPS) is calibrated yearly to maintain traceability according to ISO 17025, the real measured dew point temperatures may include potential error as well as have problems of compensation process. This suggests that the effect of connection line material in the dew point measurement system should be considered and the uncertainty contribution needs to be investigated.

In this article, the calibrations for six different capacitive-aluminum oxide dew point sensors were performed for various tube material to examine differences among measured dew point temperatures and estimate measurement uncertainty between -60 ℃ and 10 ℃, reference dew points. The purpose of this paper is to analyze the effect of the tube material connecting capacitive-aluminum oxide dew point sensors and dew point calibration system on the dew point measurement. Also, we studied correlation between tube material effect and measurement results related to respective reference dew points and application method of tube material effect in the measured dew points.

EXPERIMENTAL

The effect of tube materials on measurement results was studied by the calibration of six sampling DPSs as the experiment method according to four different types of tube materials over one year. The calibration is the process of comparing the results of the instrument to be calibrated and a traceable reference standard. We performed the calibration of capacitive-aluminum oxide DPS due to four different kinds of tube materials in a calibration standard room to reduce the other uncertainty contributions as much as possible. Also, the same calibrations were performed for six commercial aluminum oxide sensors to evaluate accurately the influence due to gas lines of various materials by obtaining reliable and accurate measurement results. We chose commercial sensors of capacitive- aluminum oxide type as the instrument to be tested because those are the most widely and frequently used sensor type in industry.

The calibration system of DPS was consisted of a dew point generator that was kept constant temperature, 35 ℃, pressure swing dryer and traceable chilled mirror hygrometer as a reference equipment. Figure 1. shows the experimental configuration for the calibration of dew point temperature. The reference humid air gas was continuously supplied to the sampling DPS, capacitive-aluminum oxide type, and reference DPS, chilled mirror type, through a dew point generator connected with a pressure swing dryer. The sampling DPS and reference DPS were connected by tubes to the gas outlet of each dew point generator. The total gas flow rate inner calibration system was controlled by two mass flow controllers, MFC_1 and MFC_2. One flow by the MFC_1 was introduced to reference standard. The other by the MFC_2 was introduced to sampling DPS. The flow rates to sampling DPSs and reference standard DPS were controlled respectively with 3 L/min and 0.5 L/min under the standard condition of 101.325 kPa in all the measurement. The connection lines were made of stainless steel, PTFE, copper and PVC with an inner diameter of 4 mm and a length of 1500 mm. All the four connection sections between reference standard and sampling DPS were prepared of almost the same length. Basically, the whole calibration system included in dryer, generator and reference DPS were connected with stainless steel, whereas the connection of sampling DPS and generator was used separately with four different material tubes. The variation tendency of corrections and differences among the measured dew point temperatures due to lines with four different materials-stainless steel, PTFE, copper and PVC at each reference dew point, were analyzed and measurement uncertainties in each case were evaluated. The stainless steel, PTFE, copper and PVC are typically used as gas line material of dew point measurement system in the field, however, the measurement error due to line materials have not been considered.

The calibrations were conducted at constant room temperature, (23±0.2) ℃ and a pressure of 1 bar. To prevent condensation during measurement, the temperature of all the connection structures including the calibration system and hygrometer were kept above the reference temperature.

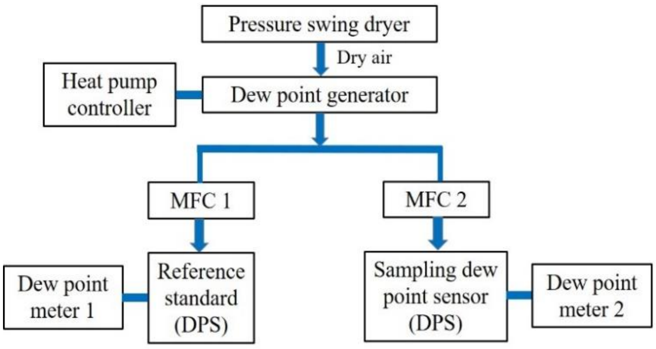

Figure 1: Experimental Configuration for the Investigation of Dew point difference between Reference Standard and Sampling DPS: Reference Standard-chilled Mirror Sensor, Sampling DPS-capacitive Aluminum Oxide Sensor.

For this, the connection section of calibration system was heated to a temperature about 20 ℃ higher than the reference temperature before setting the reference dew point. In addition, a leak test was performed to prevent leakage due to defects in the system. The purge operation was conducted for about 13 hours before performing the calibration in order to minimize the residual moisture of the entire calibration system. The calibration was performed at a reference temperature of -60 ℃ to 10 ℃ according to standard calibration method of Korea Testing Laboratory recognized by ILAC (International Laboratory Accreditation Cooperation) and KOLAS (Korea Laboratory Accreditation Scheme). The estimation of measurement uncertainty was also based on the documents EA-4/02 *Expression of the Uncertainty of Measurement in Calibration* [19].

RESULTS AND DISCUSSIONS

Effect of Various Tube Materials on Dew Point Temperature

We examine the effect of various tube materials, stainless steel, PTFE, copper and PVC on the measurement results according to reference dew points using the calibration system. To investigate experimentally, the correction and uncertainty contribution related to the tube materials, we analyzed the measurement results by reference standard and the sampling dew point sensors. The average of three measurement results for six aluminum oxide sensors were calculated due to the change of four materials of tube between -60 ℃ and 10 ℃.

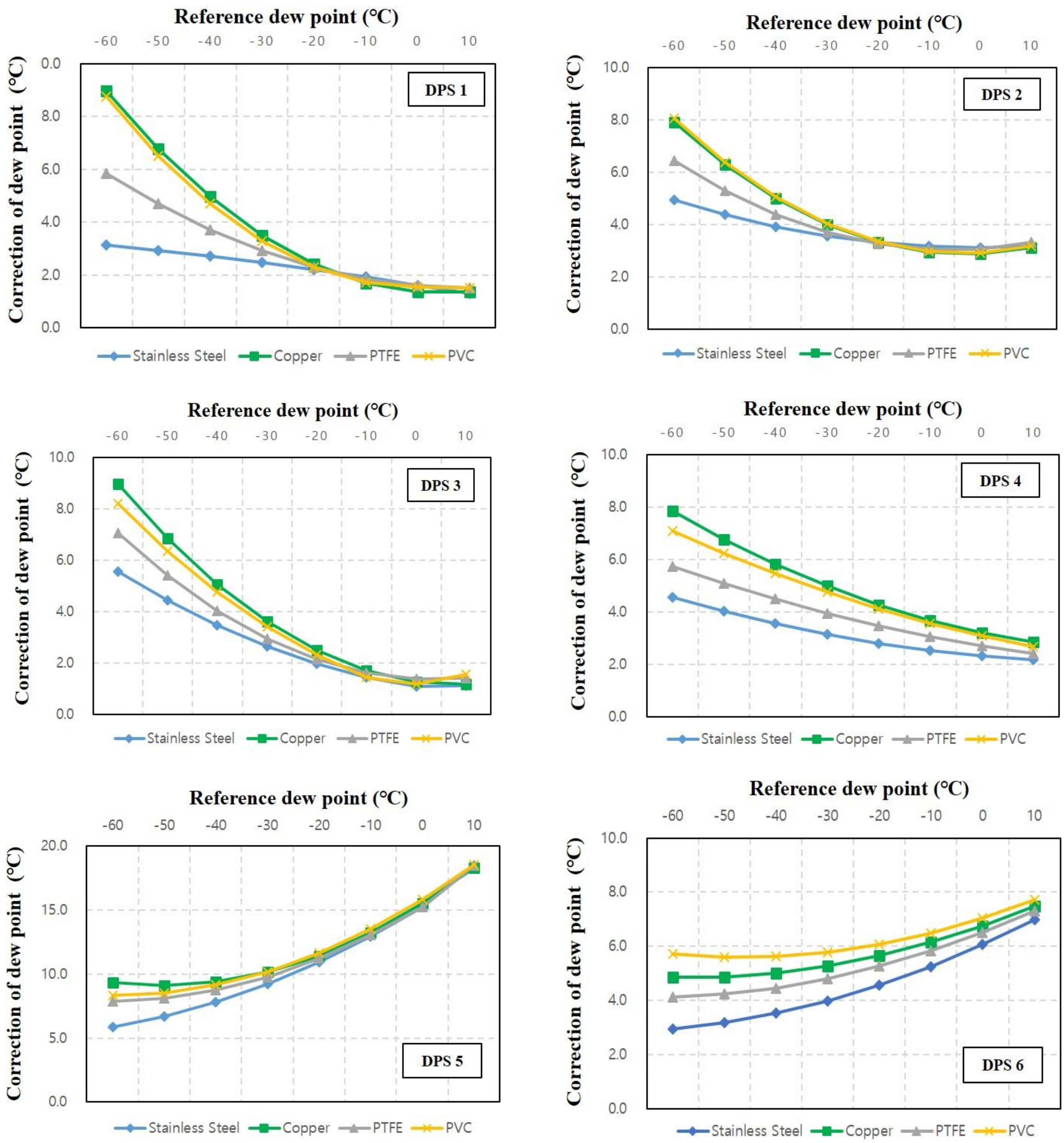

Figure 2: Variation of Correction Values Obtained with four different Tube Materials under Reference Dew Points.

Figure 2: shows the respective variance of correction values for four different tube materials under reference dew point. The quantitative correction values based on reference dew points were much different among six sampling DPSs. However, the analysis of changing trend of correction relevant to tube materials in order to evaluate individual magnitude of effect due to variation of reference values is more important than the real quantitative deviation which may be caused by the characteristic and previous adjustment of sampling DPSs. There is little difference among the trend of correction patterns due to reference dew points in all the experiments. There were somewhat differences of the corrections due to the sampling DPSs based on variation of connection tube materials. However, the corrections of dew point in case of the stainless steel tube were smaller than those in the others, whereas the corrections in case of the copper tube were relatively larger than those in the others on all occasions. Also, the deviation of correction values increased generally as the reference dew point decreased. In particular, the variance rate of them increased sharply at reference dew point below -20 ℃, relative humidity about 3 %rh at 23℃, reference temperature. It is indicated that the lower the reference dew point, the greater the influence of tube material on the measured values on account of sensitivity for subtle variation of the residual moisture existed in moist air in the range of low humidity. The deviation of correction values, conversely, became smaller as the reference dew point temperature increased. There were little differences in the correction values regardless of the influence of the tube material above the reference temperature of -20 ℃. The tube material the greatest affecting the measurement results is the copper, followed by PVC and PTFE compared with the stainless steel representing the lowest correction in below approximately -20 ℃, reference dew point.

Analysis of Correlation between Tube Material and Reference Dew Point

In this section, we evaluated the differences between maximum and minimum values among measured dew points in the calibration system applying four kinds of tube materials with respect to each reference dew point. The analysis of measured dew points was performed for six sampling DPSs. Figure 3 shows the deviation of measured values according to the change of tube material at each reference temperature and the rectangle boxes in each graph are the range of measurement uncertainty (k=2) excluding the tube material effect. The differences of the results due to four kinds of tube material were presented differently according to reference dew points. In particular, the highest deviations were recorded at reference dew point, -60 ℃ for all the DPSs. The deviations between maximum and minimum values among measured dew points related to tube material effect at -60 ℃, -50 ℃, -40 ℃ (see Figure 3 (a), (b), (c)) were above approximately 3 ℃, 2 ℃ and 1.5 ℃. These results were the numerical values exceeding respective measurement uncertainty.

Figure 3 ((d), (e), (f), (g), (h)) show the deviations of measured dew point influenced by tube material effect in the range between -30 ℃ and10 ℃, reference dew points. It shows that the maximum difference of the results was recorded between 1.9 ℃ and 0.2 ℃. There were small deviations relatively in the analysis results regardless of the tube material types as well as those existed within the measurement uncertainties. In particular, the deviations at above 0 ℃, reference dew point, were present under 1 ℃ on all occasions. As expected, there was little effect of the tube material on the calibration results above the reference dew point of approximately 0 ℃.

Change of the deviations for individual DPS in the range of below -20 ℃ was bigger according to reference dew points while that of the deviations was relatively constant in the range of above -20 ℃. The average values of measured dew point deviations obtained from the six sampling DPSs due to the tube material effect at each reference dew point is presented in table 1. The average deviation of measured dew point at -60 ℃ was approximately five times as high as that of measured dew point at above -20 ℃, reference dew point. These results suggest that the tube material effect appears differently according to reference dew point and the tube material has a great effect on the accuracy and reliability of

measurement result in lower moisture contents with low dew point temperature.

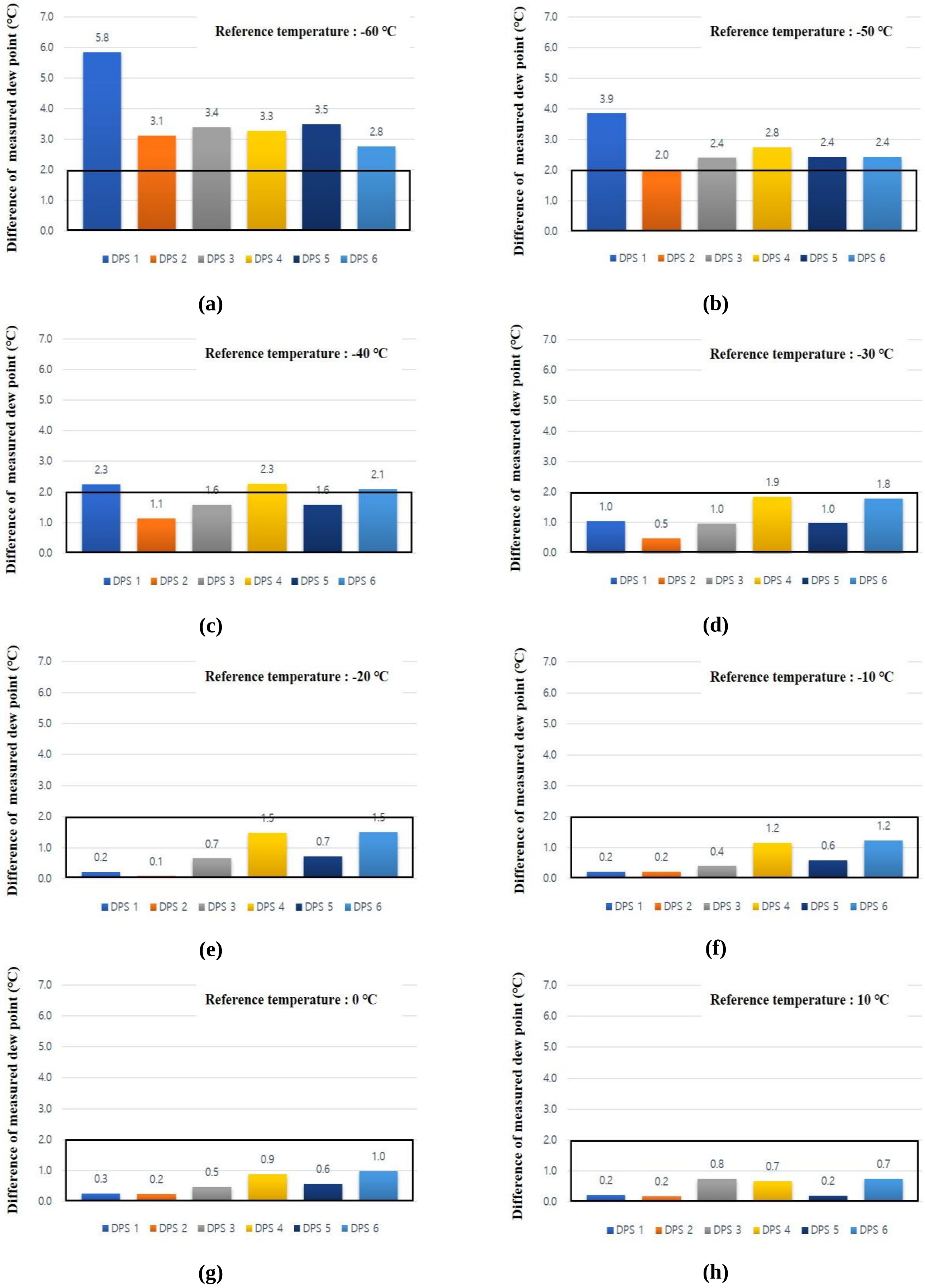

Figure 3: Comparison of Differences Between Maximum and Minimum Measurement Values due to Four Kinds of Tube Materials at Respective Reference Dew Points. Rectangle Box Show the Range of Measurement Uncertainty (K=2) Excluding the Effect of Tube Material.

Table 1: Average Deviation of Measured Dew Points Due to Various Tube Materials

Reference Dew point (℃)	Average Deviation (℃)
-60	3.6
-50	2.6
-40	1.8
-30	1.2
-20	0.8
-10	0.6
0	0.6
10	0.5

The difference caused by the characteristic of tube material could be regarded as a measurement error. The measurement conditions such as reference instrument, purging rate, total flow rate and connection tube length were maintained identically during the measurements for the all sampling DPSs. Hence, difference values of measured dew points probably originated in the residual moisture by adsorption rate related to the respective tube material. As the reference dew point increased, the difference of measurement result by the effect of tube material decreased generally, to greater or lesser degrees. From these results, it was found that the adsorption of residual moisture inside the total calibration system negatively affected the measurement results. Namely, the unstable residual moisture in air flow due to adsorption on the connected tube surface could be caused measurement error that need to be dealt with as uncertainty component. It is desirable that the effect of tube material on measurement results should be considered because the deviations of measured dew points occurred by the tube materials depended on reference dew point as well as were out or near of the limit of those measurement uncertainties in below -20 ℃. Also, even if the effect of tube material could have relatively low impact on the results in high dew point temperature, the tube material effect canbe relatively important as the contributions of other uncertainties decrease in recent years.

Uncertainty Contribution Related to Tube Material Effect

The dew point temperature of capacitive- aluminum oxide sensor is determined from the structural response of capacity values related to variation in the degree of moisture condensation in the air. Therefore, it is necessary to remove the residual moisture inside the entire system as much as possible before performing measurement as well as control not to allow any moisture to enterin humid air on measuring. The mechanical characteristic in terms of adsorption rate due to material types has been already studied [4-7]. However, the effect of material feature on dew point measurement have not yet identified clearly. In addition, tube material is being used randomly in the fields with no consideration for application method of uncertainty or compensation for measurement error occurred by material feature. Therefore, it is necessary that the effect of tube material on measurement uncertainty to obtain reliability of measured dew points at respective reference points.

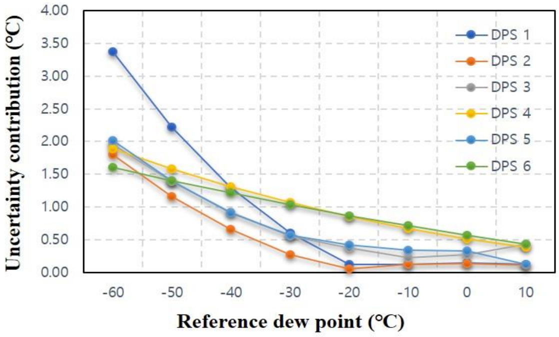

Figure 4: Comparison of Uncertainty Contribution for Four Different Tube Material Effect of Sampling Dew Point Sensors.

The variation trend of uncertainty contributions relevant to reference dew points was investigated to evaluate the effect of tube material on the measurement uncertainty. We evaluated the measurement uncertainties including tube material effect as an uncertainty component. The uncertainty evaluation method was based on the international standard EA [18] and the uncertainty related to the experimental repeat measurement was determined using the A type evaluation, and the uncertainties related to the reference instrument, resolution, stability and fitting were determined using the B type evaluation. The uncertainty contribution to the standard uncertainty of the tube material effect was estimated from the deviation calculated in this study using type B evaluation method [18-21]. Figure 4. presents the uncertainty contributions for different tube material effect as an uncertainty component. Because the effect of tube material was investigated by calibration for six dew point sensors, the previous results was applied for estimation of uncertainty contributions. The uncertainty contributions for tube material effect as an uncertainty component were the highest estimated at -60 ℃, reference point while those were the lowest estimated at 10 ℃, reference point for all the sensors, the contribution behaviors as a whole had a similar pattern. The lower the reference dew point, the more tube material effect has an influence on the contribution to the standard uncertainty.

Table 2: The Measurement Uncertainty Budget at Reference Dew Point Temperature -60 ℃ (Excluding Tube Material Effect)

Uncertainty component	Standard uncertainty	Distribution	Sensitivity coefficient	Uncertainty Contribution	Degree of Freedom
Repeated measurement	0.37 ℃	T	1	0.37 ℃	6
Standard calibration certificate	0.07 ℃	Normal	1	0.07 ℃	∞
Influence of long term stability for standard	0.06 ℃	Rectangular	1	0.06 ℃	50
Influence of standard correction	0.01 ℃	Rectangular	1	0.01 ℃	∞
Resolution of measurement	0.03 ℃	Rectangular	1	0.03 ℃	∞
Curve fitting for correction	0.87 ℃	Rectangular	1	0.87 ℃	6
Stability of generator	0.06℃	Normal	1	0.06℃	50
Distribution of generator	0.00 ℃	Rectangular	1	0.00 ℃	50
Combined standard uncertainty				0.96℃	22

The quantitative contribution values were different somewhat in the range below -20 ℃, depending on the sensors' manufacturers and brands, however, those were less than approximately 1 ℃ and 0.5 ℃ respectively in the range of reference dew point between -20 ℃ and 0 ℃ and above 0 ℃. In particular, the slop of variances for the contributions increased sharply on all occasions as the reference dew point decreased from -20 ℃. It is assumed that the extremely small unequal residual moisture adsorbed on the connection tube surface make the generated reference gas's dew point temperature unstable.

The selecting uncertainty components and evaluating measurement uncertainties for dew point sensor calibration were based on statistical theory and experimental results (table 2.) This budget based on format given in the document "Expression of the Uncertainty of Measurement in Calibration" [18] is the typical measurement uncertainty in terms of calibration of the sampling dew point sensor to compare the uncertainty including the tube material effect. The measurement uncertainties evaluated in this paper are calculated under the normal distribution of the coverage factor, k=2 at confidence level of approximately 95 %.

The comparisons of measurement uncertainties applying the uncertainty contribution for tube material effect are presented in Figure 5. Although the rates of change of measurement uncertainties including the influence of tube material according to the reference temperature were different, it was found that in all experiments, the measurement uncertainty rapidly decreased as the reference dew point temperature increased.

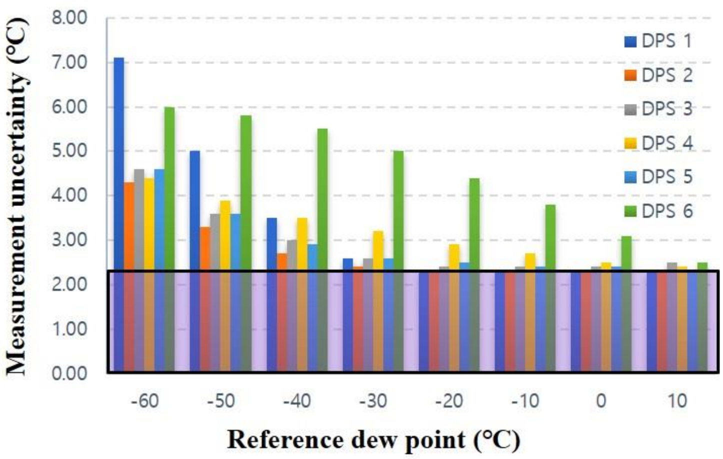

Figure 5: Comparison of Measurement Uncertainty related to Reference Dew Point Temperature. Rectangle box show the Range of Measurement Uncertainty (*k*=2) Excluding the Effect of Tube Material.

Also, although there might be differences depending in the sensor, the uncertainty including the influence of the tube material was almost the same as the uncertainty without it at the reference dew point of 0 ℃ or higher. In other words, it can be judged that the uncertainty contribution to the tube material effect is insufficient as the reference dew point temperature increases. The maximum deviation values of the sampling DPSs were from 2.1 ℃ to 5.0 ℃at the reference dew point of -60 ℃.

From this result, it is expected that the contribution of uncertainty due to the influence of the tube material as the uncertainty component in the dew point temperature range below -60 ℃ is considerably large. In particular, there was little effect of tube material on measurement uncertainty in the reference dew point region of approximately 0 ℃ or more. The

evaluation of the standard uncertainty with respect to the tube material under low moisture content below the reference temperature of 0 ℃ is more important than that of under high moisture content. It is judged that this is because the sensitivity of the material to the uncertainty is greater than that of other uncertainty components at a minute moisture content. Therefore, it is desirable to identify the characteristics of the tube material at a low reference dew point temperature of 0 ℃ or less and reflect it in the measurement uncertainty evaluation.

CONCLUSIONS

In this study, the effects of tube material on dew point measurement and measurement uncertainty was investigated by experimental and statistical techniques. The investigation was focused on variances of correction and deviation of measured dew points indicated by six dew point sensors related to connection tube with stainless steel, copper, PTFE and PVC in the reference range between -60 ℃ and 10 ℃.

The correction values were found to increase with decreasing generated reference dew point temperature and the variance ne rate increased sharply in the area below reference dew point of-20 ℃. The deviations of measured dew points by the tube material effect decreased generally, to greater or lesser degrees, when the reference dew point increased. The effect of tube material at reference point with -60 ℃ was approximately five times as high as that of tube material at above -20 ℃. The tube material the greatest affecting the measurement results was the copper, followed by PVC, PTFE and stainless steel in the range of reference dew point below approximately -20 ℃. The tube material effect had a great effect on the accuracy and reliability of measurement result in the lower moisture contents.

To investigate the effect of tube material on the measurement uncertainty (k=2), uncertainty contributions related to tube material effect as an uncertainty component for six dew point sensors evaluated as well as the measurement uncertainties including the contributions of material effect were compared with those excluding the contributions. The uncertainty contributions were estimated the highest at -60 ℃, whereas those were the lowest estimated at 10 ℃ in all the cases. The evaluation of the standard uncertainty with respect to the tube material under low moisture content below the reference temperature of 0 ℃ is more important than that of under high moisture content. The contributions to the standard uncertainty related to tube material effect in the low moisture condition were more significant compared with that in the high moisture condition. In particular, it can be seen that the uncertainty contribution related to the effect of the tube material on the total uncertainty exists as a domination term below 0 ℃.

It is necessary to grasp the correlation between tube material effect and measured dew point related to respective reference dew points and application method of measurement uncertainty to improve the reliability and quality of measurement results. Also, it is desired that effective compensation method considering the effect on the dew point according to tube materials should be established in the field.

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